INVESTIGATING DIELECTRIC PROPERTIES OF SINTERED POLYMERS FOR RAPID MANUFACTURING

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Abstract

Selective Laser Sintering (SLS) of polymers is the leading technology in the growing field of Rapid Manufacturing. High Speed Sintering (HSS) is a process that offers the potential to reduce costs and processing times and thus open significant new markets for Rapid Manufactured parts.

Much academic research has been performed with respect to mechanical properties of Rapid Manufactured parts, however the area of electrical properties has received little attention to date. Electrical properties are obviously important in applications that will involve embedding of circuits with Rapid Manufactured 3D objects. However electrical properties are also important for a wide variety of electrical products where Rapid Manufactured parts can be used as housings etc.

This paper focuses on the dielectric properties of parts made by SLS and HSS and compares properties with those for conventionally processed polymers. Dielectric strength results show that SLS parts are comparable with injection moulded parts, while HSS parts are inferior to SLS parts. Dielectric constant and dissipation factor results show that HSS parts are comparable with injection moulded parts, whilst SLS parts have superior properties. The presence of porosity (SLS and HSS) and the presence of carbon (HSS) are suggested as reasons behind the variation in dielectric properties when compared with injection moulded parts.

1 Background

1.1 Rapid Manufacturing

Selective Laser Sintering (SLS) of polymers is the leading technology in the growing field of Rapid Manufacturing (RM). This is partially due to the high mechanical properties of parts that it produces, as well as the stability of polymers which can be processed. Nylon-12 is one such polymer, and is the most common to be processed via SLS.

High Speed Sintering (HSS) is an emerging layer manufacturing technique, aimed at producing end-use parts. The HSS process has similarities with SLS in that it produces robust parts from polymer powder. However, to compete in the field of RM, HSS has several key advantages, including potentially lower machine costs and faster build speeds [1]. These advantages are as a direct result of using the combination of an infrared (IR) radiation source to initiate sintering (which is less expensive than a laser system), coupled with an inkjet system to define the 2D area to be sintered (which is quicker than the scanning process of a laser).

The layer cycle in HSS begins with the deposition of a layer of nylon powder onto the part bed. Currently this is achieved with a roller assembly moving from left to right, using the apparatus shown in Figure 1. A radiation-absorbing material (RAM) is then printed directly onto the surface of this powder as a 2D profile which represents the area to be sintered. This involves the assembly in Figure 1 moving from right to left. As the print-head traverses the part bed, it is followed by an IR lamp which exposes the printed profile to IR radiation. When the IR radiation reaches the surface of the part bed some of the thermal energy is absorbed and some is reflected. However, the RAM, which contains carbon, absorbs the emitted radiation at a much higher rate than the raw powder in the unprinted regions [1]. This thermal energy is transmitted to the powder directly below the RAM via conduction, causing this powder to reach its melting temperature and thus initiate viscous sintering. Unprinted regions cannot absorb the radiation quickly enough to cause the powder to melt and hence remain in an unsintered state. Figure 2 shows of few examples of parts that have been produced via HSS.

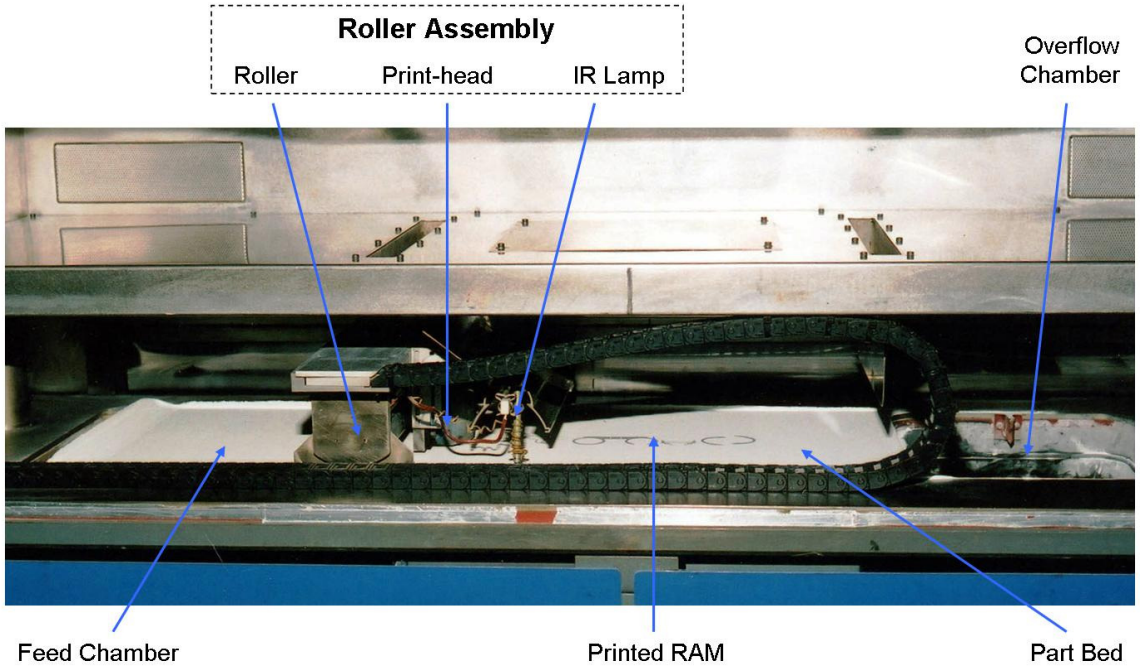

Figure 1 – Picture of the HSS apparatus

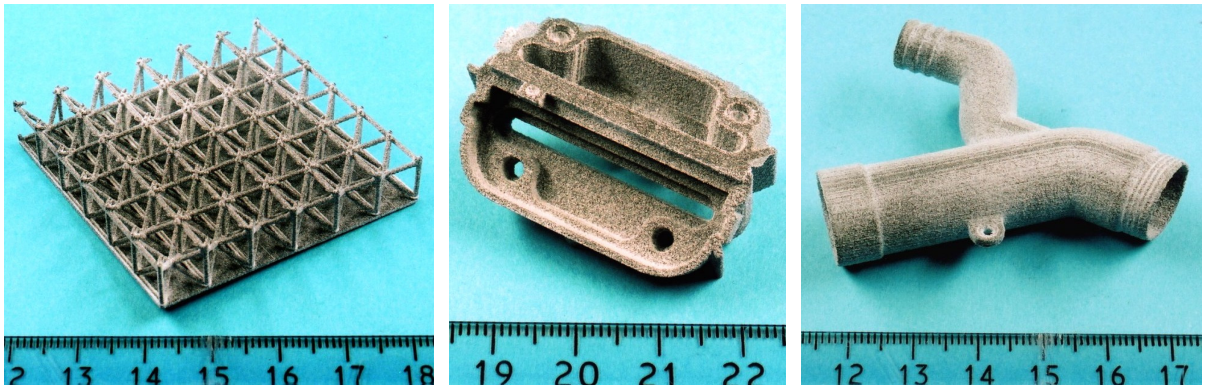

Figure 2 – Examples of HSS parts

1.2 Electrical and Electronic Applications

In addition to SLS and HSS there are several other processes in existence or under development in the growing field of RM. As well as recognising the potential benefits of producing end-use parts via these technologies, RM researchers are also beginning to explore new applications and niches that they can branch into. One area pertinent to this paper is that of electrical and electronic products, where there exists a need to produce parts which have integrated electrical and mechanical functionality.

Historically, when only Rapid Prototyping technologies were considered, there was no real need to prototype 3D electromechanical assemblies via layer manufacturing as ultimately their manufacture as end-use products would have been via conventional processes. However, the realisation of RM now means that there is a drive to use these technologies to produce such products, and bring the many inherent benefits of layer manufacturing to them. Examples of research performed in this area include the fabrication of electrical interconnections [2], the integration of direct-write technology into an RM environment [3], and the fabrication of functional parts such as piezoelectric components [4], zinc-air batteries [5, 6], polymer-based actuators [7, 8], electromechanical relays [9] and electrochemical transistors [10].

1.3 Dielectric Properties

For integrated electromechanical parts to be realised using RM, a significant amount of research is required into materials as well as processes. In addition to the development of electrically conductive materials (e.g. for the fabrication of electrical interconnections), there is also a need to establish suitable electrical insulators as any electrically/electronically functional components will need to be protected and integrated. The electrical properties of insulators (often referred to as dielectric properties) are also important to a wide range of electrical products, where RM parts may be used as components or housings. There are several different dielectric properties that can be used to quantify the dielectric performance of a material and, depending upon the application, some are more important than others. Figure 3 shows some of the most important properties and the applications for which they are most significant.

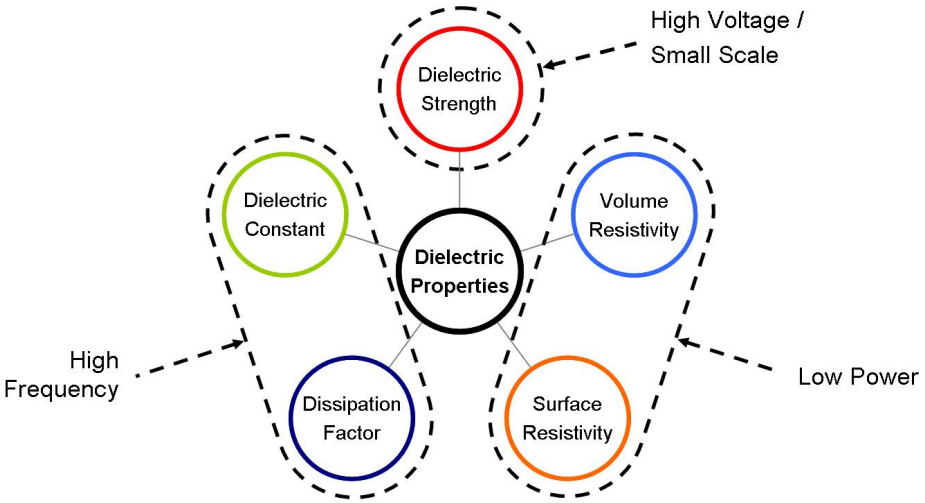

Figure 3 – Key dielectric properties and applications to which they have most significance

As discussed, there has been a significant amount of research performed to investigate the feasibility of using RM technology to produce electrical and electronic parts, mainly focussed around process development and electrically conductive materials. A limited amount of research has been performed to investigate the dielectric strength of certain SLA materials [11], however this work only attempted to characterise their performance and does not offer explanations as to the possible causes of variation in dielectric performance between materials.

Hence, the authors have identified that there exists a need to investigate the dielectric properties of RM materials, and to understand how they are influenced by composition and processing. To a designer, this information is critical as it cannot be assumed that RM materials will behave like conventionally processed materials. The remainder or this paper presents the authors' initial research into the dielectric properties of sintered nylon-12 parts produced via SLS and HSS. In particular, dielectric strength, dielectric constant and dissipation factor were the focus of this work.

1.3.1 Dielectric Strength

Dielectric strength is a measure of the maximum electrical field strength that a material can withstand, after which electrical (dielectric) breakdown occurs. In polymers, this failure is usually in the form of a carbonised puncture through the material, and as a result the material permanently loses its dielectric properties. Numerous breakdown mechanisms have been hypothesized in the literature for dielectric breakdown [12-14] and these can largely be grouped into electronic, thermal and electromechanical mechanisms. However, the actual breakdown of a material tends to be as result of a combination of these mechanisms [14], which makes the specific mechanism responsible for initiation of breakdown in a test sample difficult to determine. A high dielectric strength is almost always desirable for an insulating material.

An obvious area in which dielectric strength is important is in high voltage applications, where the electrical fields can be high enough to cause dielectric breakdown through a significant thickness of material. However, as electronic assemblies continue to increase in complexity, there is a drive towards miniaturisation and integration. This means that the dielectric strength of an insulating material can also be important for lower voltage applications, where spacing between electrical elements continues to reduce.

1.3.2 Dielectric Constant and Dissipation Factor

When a material is exposed to an electrical field it will absorb some of the electrical energy (i.e. polarization). The dielectric constant can be used as a measure of how much energy is stored by the material, and is a ratio of the material's capacitance to the capacitance of a vacuum of the same volume. For most applications (with the exception of capacitors and memory cells) dielectrics with a low dielectric constant are required as this helps to minimise interference problems such as crosstalk between interconnections, reduce energy consumption and signal propagation delays, and maximise bandwidth.

The dissipation factor represents the energy loss from the absorption process, and is a ratio of the energy which is dissipated to the energy which is stored in the material. Dissipated energy typically turns into heat or is radiated into the materials surroundings at radio frequencies

(RF). To maximise efficiency and prevent problems such as dielectric heating (high-power) or signal loss (low-power), it is usually desirable to have a material with a low dissipation factor.

2 Experimental Methodology

To measure the dielectric strength, dielectric constant and dissipation factor of SLS and HSS parts, two different types of dielectric tests were performed:

- **Dielectric breakdown testing** used to determine the d.c. *dielectric strength*.
- **Dielectric spectroscopy** used to determine *dielectric constant* and *dissipation factor* as a function of a.c. frequency.

Dielectric breakdown testing involves the gradual increase of a voltage applied to a test sample, until electrical breakdown of that sample occurs. From the measured breakdown voltage and sample geometry, dielectric strength can be calculated. Dielectric spectroscopy measurements involve the study of the electrical current response of a material to an applied electric field, from which dielectric constant and dissipation factor can be calculated [15]. For each of these tests, FIVE different samples were measured for each RM process. Details of the specific test methods are discussed in more detail later in this section.

2.1 Sample Preparation

2.1.1 Geometrical Considerations

When measuring dielectric properties, sheet specimens are preferable as they allow for a simple parallel-plate electrode set-up to be used [16], and also simplify any calculations that need to be made based upon sample thickness or electrode spacing. The geometry of a sample is particularly important to dielectric breakdown testing as the electrical fields involved are often of a magnitude in which it is possible that electrical breakdown can occur in the surrounding medium (known as flashover), creating an electrical short around the sample as opposed to causing breakdown through it. To minimise the risk of this occurring, samples were designed to be as large as possible and thus would overhang the test electrodes.

Unfortunately, at the time of sample manufacture the HSS system was only capable of building parts in a narrow strip down the centre of the part bed, so samples could only be manufactured with a maximum width (Y) of 50 mm. A length (X) of 100 mm was chosen to allow for the samples to be easily handled, without contamination of the test area by skin oils, etc. Also, sample thickness (Z) was kept low (1 mm) so that the voltage required to initiate dielectric breakdown would be minimised and thus further reduce the risk of flashover occurring. The sample geometry is shown in Figure 4.

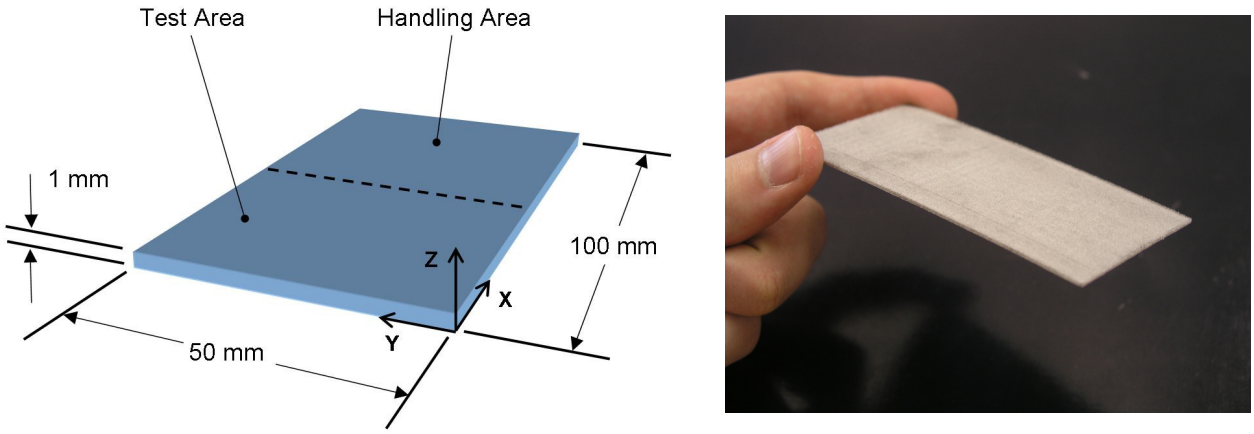

Figure 4 – Test sample geometry (left) and photo of actual part (right)

2.1.2 Fabrication

Test samples were fabricated in a single build for each process, using non-virgin (recycled) Duraform ® PA nylon-12 powder. Once built, the samples were thoroughly cleaned of unsintered powder. Typically a bead-blaster would be used for speed, however the risk of glass beads becoming embedded in the sample meant that this was not a suitable method, and so all samples were cleaned by hand only.

2.1.3 Conditioning

Any contamination on the surface of a material, even from manual handling, can affect the measured dielectric properties [16], so Isopropanol was used to cleanse the surfaces of all samples prior to testing. A sample's moisture content can also have a significant effect upon dielectric measurements, so to ensure comparable results this was controlled. Ideally samples would have been left in a temperature and humidity controlled atmosphere for 48 hours to reach equilibrium, however the unavailability of such facilities meant that this was not possible and samples were instead dried using a vacuum oven. With the oven's chamber temperature stabilised at 50℃, samples were left for 1 hour to remove any moisture and residual solvent. After this period the heating element was switched off and the samples were then left in the evacuated chamber for a further 48 hours.

2.2 Dielectric Strength Measurement

ASTM D 3755 [17] was followed in general for this testing, however smaller electrodes were used and tests had to be performed in ambient conditions rather than at controlled temperature and humidity.

2.2.1 Control of Variables

There are several key variables that can affect the measured dielectric strength of a material [17], so these were controlled to ensure accurate and comparable results would be obtained:

Test Conditions: Ideally the breakdown tests would have been carried out under controlled humidity and temperature. However, the unavailability of these facilities meant that this was not possible and testing had to be performed in ambient conditions. Owing to the short test duration, all of the samples were tested within one hour and any variations in ambient conditions within this period were deemed to be insignificant.

Voltage Waveform: The voltage waveform (a.c./d.c) to be used is typically selected based upon the intended end-use of the sample material. However, the purpose of this testing was to provide an initial comparison of SLS and HSS samples with no specific application in mind, so a d.c. waveform was used due to equipment availability.

Voltage Ramp-rate: Some of the mechanisms which initiate dielectric breakdown are time-dependant, so the rate at which voltage is increased is important. In accordance with ASTM D 3755, a short-time (rapid-rise) ramp-rate of 500V/s was used.

Surrounding Medium: The medium surrounding a sample can influence the uniformity of the electrical field which it is exposed to. Partial electrical discharges can occur in this medium, which may initiate/accelerate dielectric breakdown of the test material. Due to the porous nature of the sintered samples, air was used as the medium rather than oil.

Electrodes: The electrode geometry and material also influences the electrical field that a sample is exposed to. The grounded electrode used was a Ø 25 mm brass cylinder with a 2 mm radius on its contact edge, and the high-potential electrode was a Ø 14 mm brass cylinder, surrounded by an acrylic electrode guard. In this set-up the high-potential electrode was located coaxially with the ground electrode, central to one half of the sample (Figure 5).

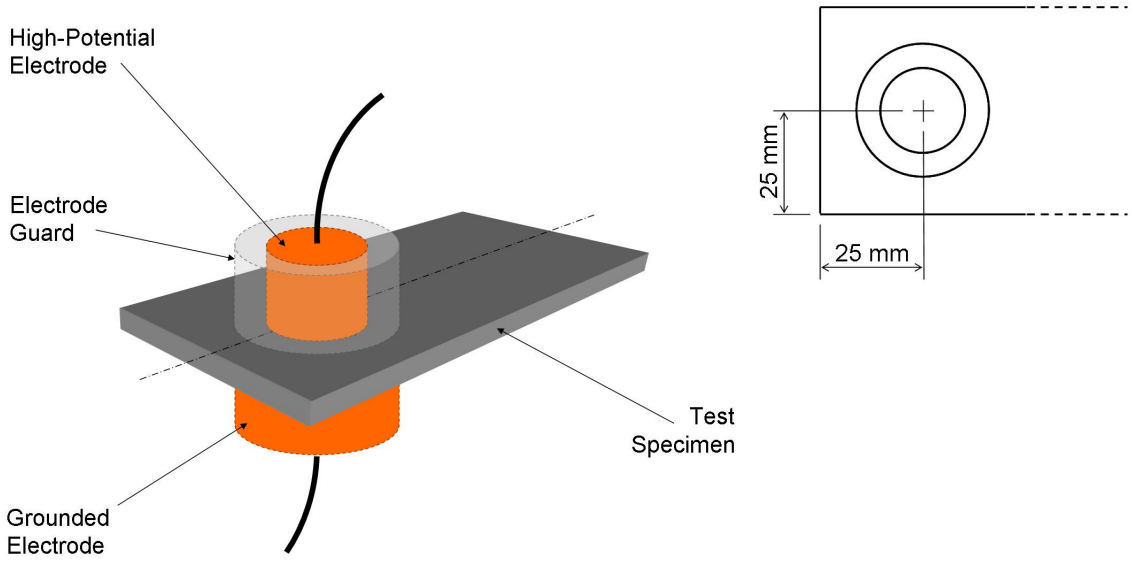

Figure 5 – Electrode arrangement for the dielectric strength measurements

2.2.2 Experimental Procedure

Dielectric strength measurements were carried out using a 100kV d.c. power supply, with a remote control panel for voltage adjustment and measurement. The following procedure was established and adhered to for all samples:

1. The test sample was first placed between the test electrodes, with the top of the sample (as it was built) facing upwards.
2. The voltage supply was switched on, with the applied voltage set to 0V.
3. The applied voltage was then increased at the predefined ramp rate of 500V/s until breakdown was initiated, evident as a rapid change in the monitored current of the test circuit (the power supply also had a built in trend /arc indicator on the remote control panel which was used to automatically detect this change).
4. Using the remote control panel's digital readout, the breakdown voltage was manually recorded and the power supply was switched off.
5. The sample was removed from the electrodes and inspected for degradation / puncturing as evidence of breakdown.
6. Using a micrometer, the thickness of the sample was measured in the vicinity of the breakdown area and also recorded.

2.3 Dielectric Constant and Dissipation Factor Measurement

ASTM D 150 [16] was generally followed for these tests, however measurements were again made in ambient humidity.

2.3.1 Control of Variables

The following variables can affect dielectric constant and dissipation factor measurements [16] and hence were controlled to ensure accurate and comparable results:

Test conditions: A Genlab oven with a Eurotherm temperature controller was used to maintain the temperature of the sample at 23℃ (to within ±0.5℃) during the spectroscopy measurements. Ideally humidity would also have been controlled, however the unavailability of these facilities meant that this was not possible and testing had to be performed in ambient humidity. Owing to the short test duration, all the samples were tested within 2 hours and any variation in ambient humidity within this period was deemed to be insignificant.

Voltage Frequency and Magnitude: The dielectric constant and dissipation factor of a polymer are frequency dependent. Therefore, measurements were made across a frequency 'sweep' from 10 Hz to 10^7 Hz. The voltage applied to the test sample was 3 V r.m.s. throughout the testing.

Electrodes: Both the grounded electrode and the high-potential electrode used were Ø 25 mm brass cylinders. In this set-up the high-potential electrode was located coaxially with the ground electrode, central to one half of the sample, as shown in Figure 6.

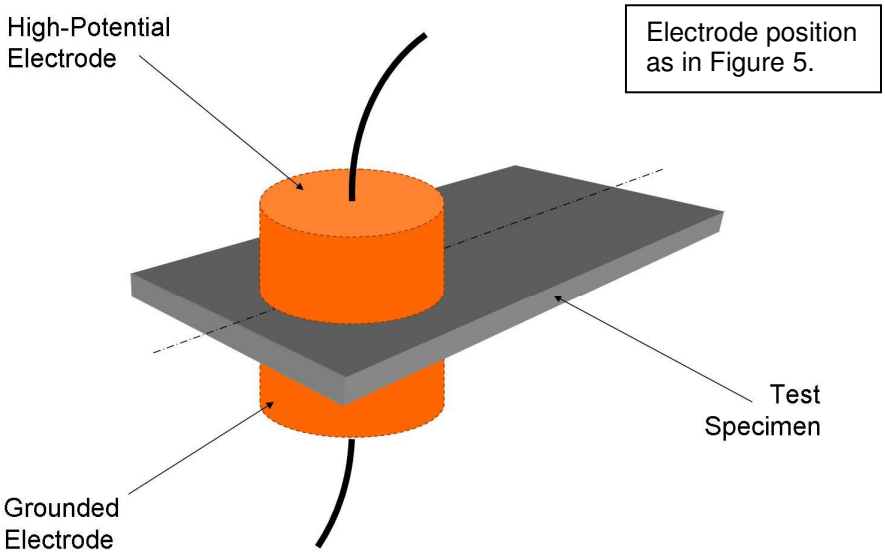

Figure 6 – Electrode arrangement for the dielectric spectroscopy measurements

2.3.2 Experimental Procedure

Dielectric spectroscopy measurements were carried out using a Solartron 1255 HF Frequency Response Analyzer (FRA), and a Solartron 1296 Dielectric Interface. This equipment was linked to a PC installed with ZPlot, a software package supplied by Solartron for configuration and control of the FRA and for the logging of measurements. By defining the electrode arrangement in this software, the dielectric constant and dissipation values were calculated automatically from the electrical current measurements. The following procedure was established and adhered to for all samples:

1. Using a micrometer, the test sample thickness was first measured in the area where the electrodes would be applied, and input into the ZPlot software.
2. The sample was carefully placed between the test electrodes in the oven, and left for 10 minutes to stabilise at 23℃.
3. Using ZPlot, the FRA equipment was initialised and measurements were logged in the software automatically.
4. Once the sweep was complete, the logged data was output from ZPlot as .CSV (comma separated values) files, ready for analysis.

3 Results and Discussion

The measured dielectric properties are reported and discussed in this section, where comparison is made between HSS and SLS results, and possible reasons for any significant variation are suggested. In addition, the results are compared with manufacturers' published figures for injection moulded nylon-12 [18], as it was not possible to determine the dielectric properties of such materials empirically. For the purposes of comparison, only rigid, unfilled and injection mouldable grades were considered, for which a total of eight were identified. These were manufactured by Arkema, EMS-GRIVORY and Evonik Degussa.

3.1 Dielectric Strength

Dielectric strength was calculated for each sample by dividing the measured breakdown voltage by the measured sample thickness. These dielectric strength results are summarised and compared with published data for injection moulded nylon-12 in Figure 7.

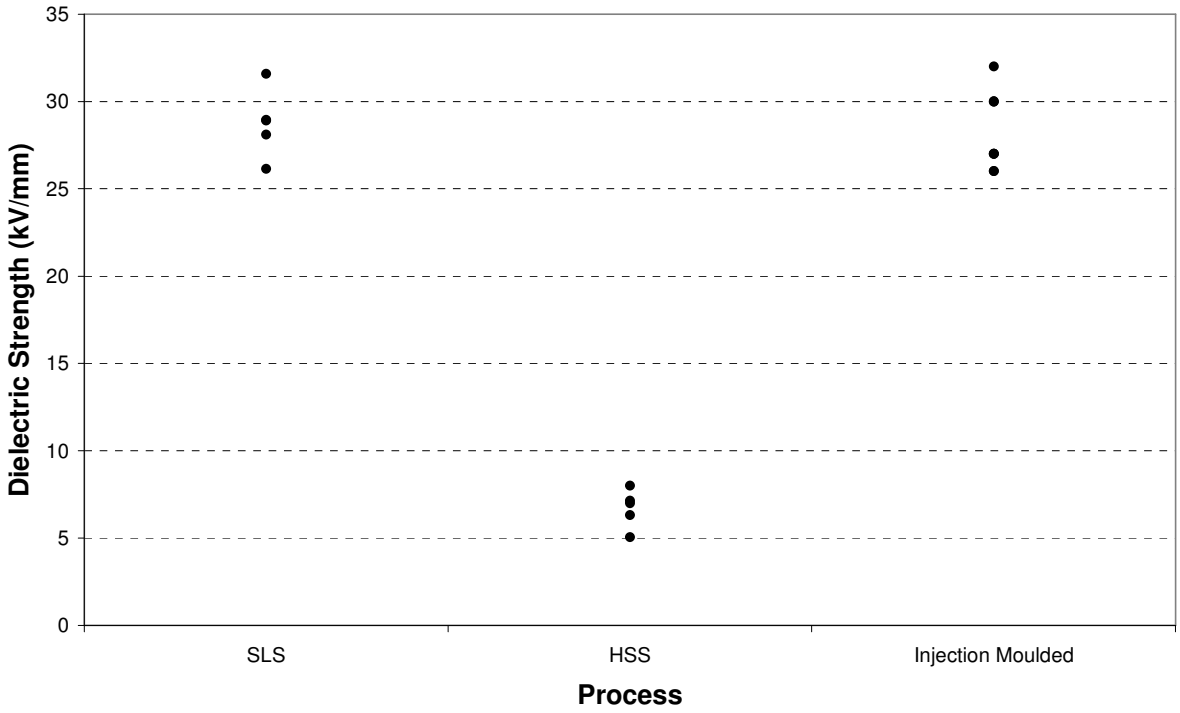

Figure 7 – Graph comparing the dielectric strength results with injection moulded nylon-12

This graph clearly shows that SLS samples proved to be superior to HSS samples by an average factor of 4. It is suggested that a possible cause of the lower dielectric strength values is the presence of RAM within the HSS samples. This agrees with other research performed on conventionally processed polymers filled with carbon-black, which identified that the presence of carbon-black resulted in a reduction in the polymers dielectric strength [19]. The exact cause of this reduction is unclear, however carbon inclusions in a material may act as stress raisers causing higher electrical fields in certain regions, thus leading to accelerated breakdown of the material.

Differences in porosity may also have caused a variation in the measured dielectric strength between SLS and HSS samples. To investigate this further, the densities of SLS and HSS parts were calculated by measuring the dimensions and masses of 5 samples for each process. These were then compared with published data for injection moulded nylon-12, as shown in Figure 8.

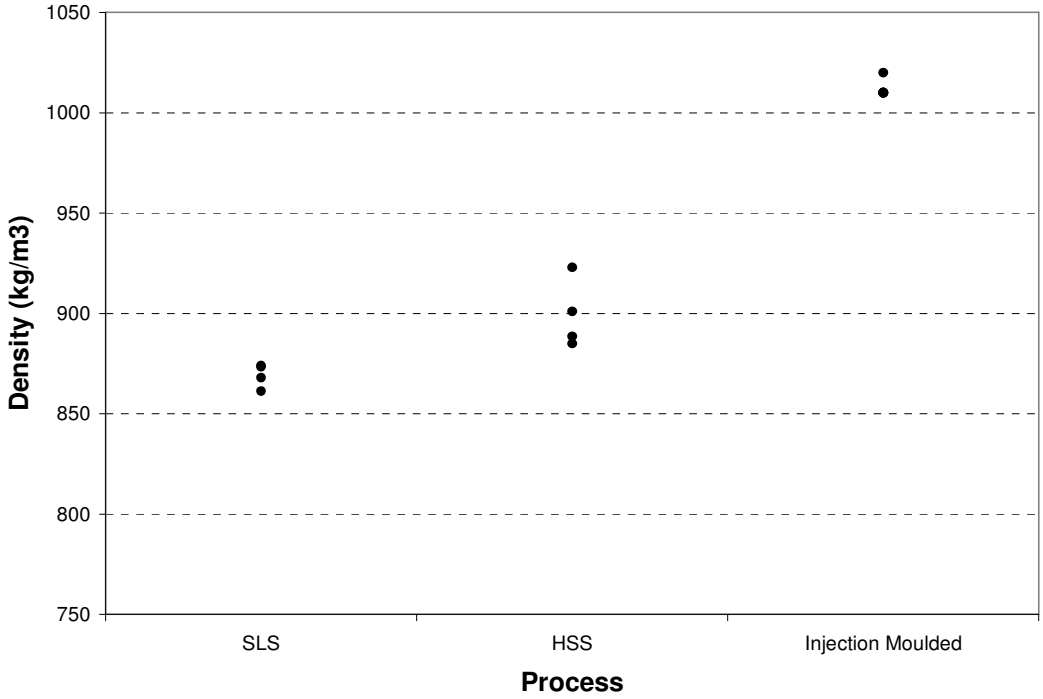

Figure 8 – Graph comparing the densities of SLS, HSS and injection moulded nylon-12

Firstly, these measurements identify that the HSS samples are marginally denser than the SLS samples, despite the fact that the HSS samples have already shown to have lower dielectric strength values. Secondly, as expected, the SLS samples have a lower density than injection moulded nylon-12, yet they have shown to have comparable dielectric strength values. Both observations support the theory that the presence of porosity in the RM samples had little effect upon their measured dielectric strengths, and the presence of carbon in the HSS samples is more likely to have caused the reduction in performance.

3.2 Dielectric Constant and Dissipation factor

Figures 9 and 10 summarise the measured dielectric constant and dissipation factor values, respectively, as a function of frequency. Range bars have been used to represent the data spread of the five samples tested for each process. Actual measurements were made up to a frequency of 10^7 Hz, however measurements taken at 100 kHz and above were extremely inaccurate due to noise, and have therefore been omitted.

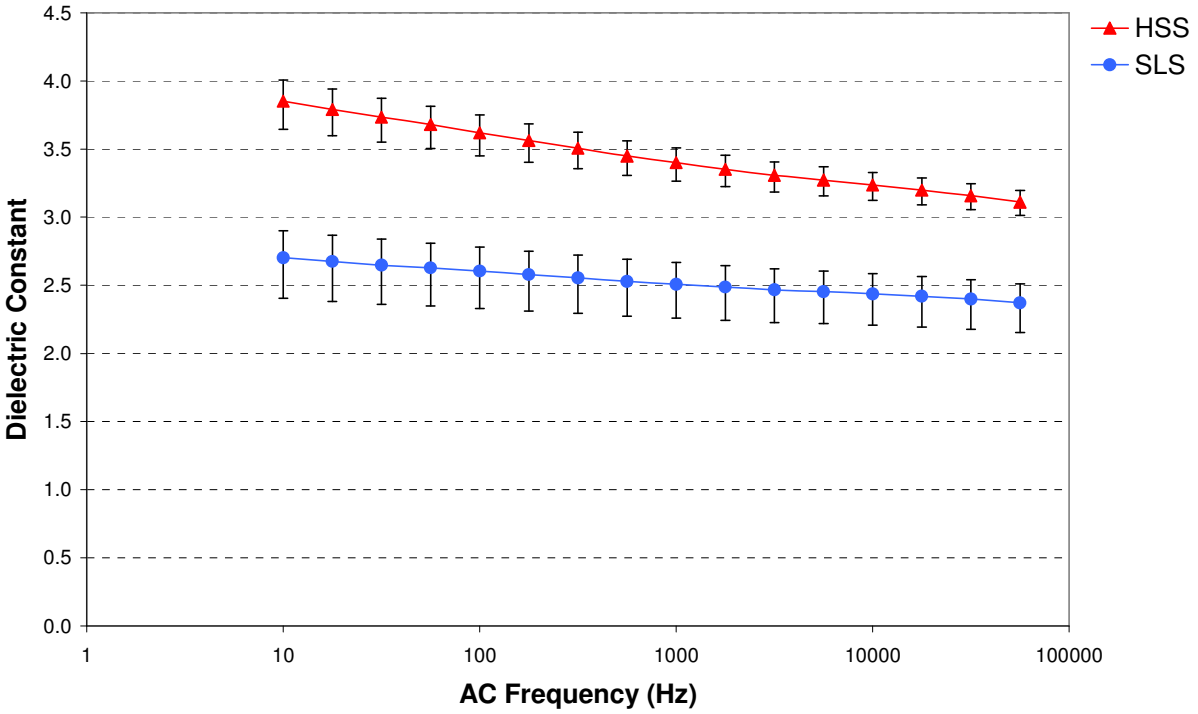

Figure 9 – Graph comparing the dielectric constant of SLS and HSS samples

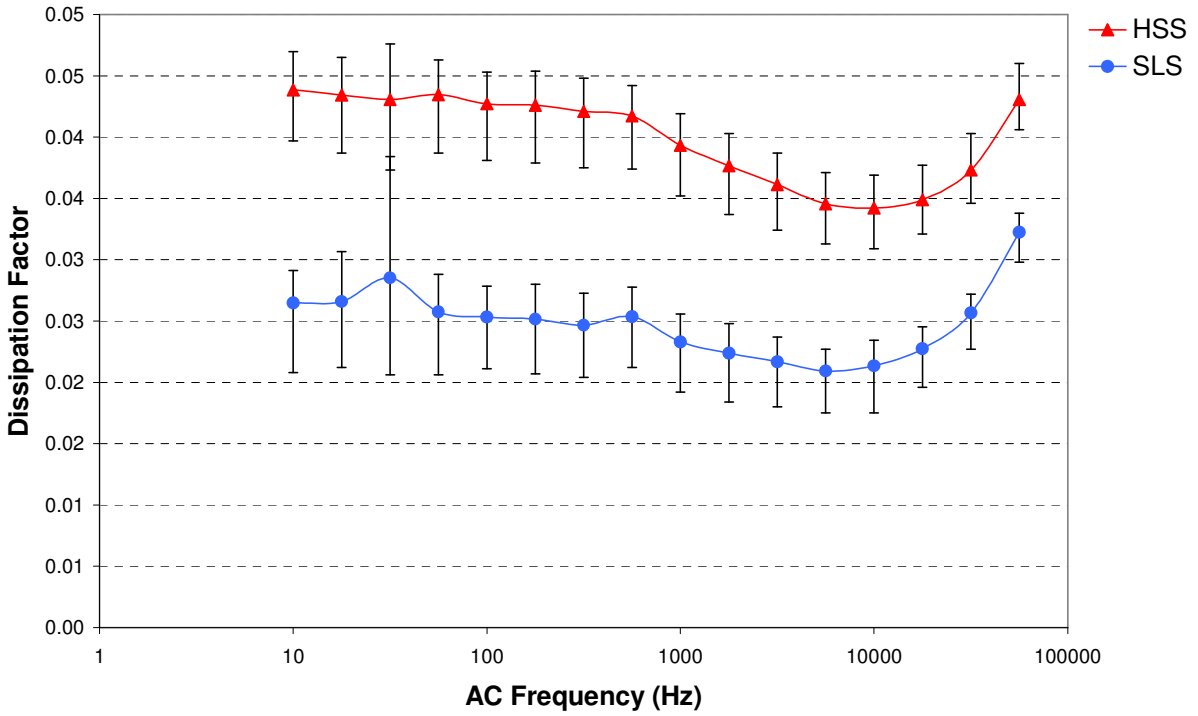

Figure 10 – Graph comparing the dissipation factor of SLS and HSS samples

These results clearly show that the dielectric constant and dissipation factor values of SLS samples were lower than, and thus superior to, those of the HSS samples. As for the dielectric strength results, it is postulated that this variation in performance is likely to be as a result of the

presence of carbon in the HSS parts. In fact, carbon is often used as a filler in applications where a material is required to have a higher dielectric constant (and sometimes dissipation factor) [20]. In both cases, SLS and HSS samples appear to respond similarly to variation in frequency, with SLS having a slightly more stable dielectric constant across the measured frequency range.

When the spectroscopy results are plotted together with published data for injection moulded nylon-12 (as shown in Figure 11), SLS samples appear to have superior performance to injection moulded nylon-12. The HSS results, although inferior to the SLS results, are comparable with injection moulded nylon-12.

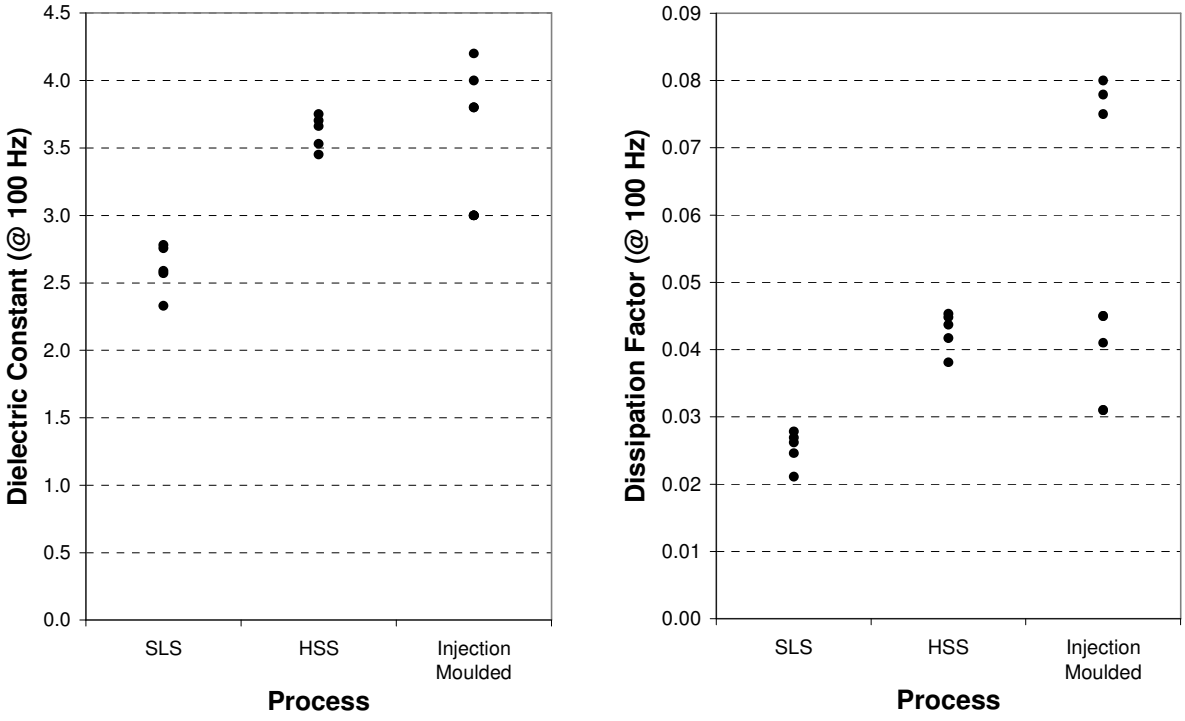

Figure 11 – Graphs comparing the dielectric constant (left) and dissipation factor (right) results with injection moulded nylon-12

It is hypothesised that the improved dielectric performance of SLS samples when compared with injection moulded nylon-12 is attributable to the presence of porosity. In two-phase materials, such as SLS parts, the bulk dielectric properties of the material are dependent upon the individual properties of each phase, as well as volumetric ratio and morphology [20]. Hence, the presence of air (dielectric constant ≈ 1, dissipation factor ≈ 0) in pores results in lower overall spectroscopy results for the SLS parts. HSS parts are also porous, however the negative effect that the presence of carbon has upon these properties means that the resultant dielectric constant and dissipation factor for the HSS samples remained comparable with injection moulded nylon-12.

3.3 Improvement of Dielectric Properties

As discussed, HSS samples have exhibited inferior dielectric properties to SLS samples and it has been suggested that this reduction in performance is caused by the presence of carbon

contained within the RAM that is printed into HSS parts. This RAM content is unavoidable as it is intrinsic to the HSS build process. However, the amount of RAM deposited onto each layer can be varied, as well as the output power of the IR lamp. By reducing the amount of RAM deposited and increasing the lamp power to compensate, it may be possible to improve the dielectric performance of an HSS part without significantly affecting its mechanical properties.

4 Conclusions

The measurement of dielectric strength, dielectric constant and dissipation factor of SLS and HSS parts has shown that SLS parts exhibit better dielectric properties than HSS parts. When compared with injection moulded nylon-12, SLS parts appear to have a similar dielectric strength, but are in fact superior in terms of dielectric constant and dissipation factor. HSS parts appear to have a significantly lower dielectric strength than injection moulded nylon-12, but comparable dielectric constant and dissipation factor.

Possible reasons for this variation in properties have been suggested. The presence of carbon (HSS) appears to have a negative effect upon dielectric performance (for all measured properties). The presence of porosity (SLS and HSS) appears to have little effect upon dielectric strength, but has a positive effect upon dielectric constant and dissipation factor. In the case of dielectric constant and dissipation factor values for the HSS samples, the combined effects of carbon and porosity seem to cancel each other out, thus resulting in HSS samples being comparable with injection moulded nylon-12.

Unfortunately the lack of empirical data for injection moulded nylon-12, of the specific grade and thermal history used for the RM parts, makes it difficult to form any firm conclusions. It is therefore suggested for further work that the investigation of the thermal history of the RM parts be carried out, and subsequent testing of injection-moulded Duraform® PA parts be performed under the same test method and conditions as described in this paper. Having done this it will be possible to compare against a more accurate benchmark.

Acknowledgements

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